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Processing Eutectics in Space

by

F.C. Douglas and S.F. Galasso

Final Report

Contract No. NAS8-29669

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George C. Marshall Space Flight Center

Marshall Space Flight Center, AL 35812

UNITED TECHNOLOGIES
RESEARCH CENTER

UNITED TECHNOLOGIES

EAST HARTFORD, CONNECTICUT 06108

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UNITED TECHNOLOGIES RESEARCH CENTER

UNITED TECHNOLOGIES

East Hartford, Connecticut 06108

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REPORTED BY F. C. Douglas
F. C. Douglas

APPROVED BY F. D. Lemkey
F. D. Lemkey, Principal Scientist
High Temperature Materials

DATE 2/10/77

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Processing Eutectics in Space

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SUMMARY

The investigations of directional solidification made under Contract NAS8-29669 and subsequent modifications were designed to establish a foundation in earth-based laboratory processing in order to properly assess the opportunity of low-gravity space processing of materials by directional solidification.

The approach used in the assessment of laboratory processed materials has been the examination of the structural regularity obtained in directionally solidified eutectic alloy compositions under conditions of different sets of solidification parameters. Alloys examined which produced lamellar microstructures were Al-Cu (33 w/o Cu) and Pb-Sn (30 w/o, 38.1 w/o, 45 w/o Pb); alloys which yielded a rod-matrix microstructure were Al-Ni (6.2 w/o Ni) and Al-Ni-Mo (6.2 w/o Al, 31.5 w/o Mo, balance Ni). The alloys were solidified in ingot form under vertical directional solidification conditions with the exception of the Pb-Sn alloys which were rolled into thin sheets and zone melted horizontally to obtain thin sheets with a regular eutectic microstructure.

The examination of the Ni-Mo-Al system is discussed in this report. A complete discussion of the experiments on the Al-Cu, Al-Ni, and Pb-Sn systems under contract NAS8-29669 is reported elsewhere (Ref. 1).

The choice of the ternary system Ni-Al-Mo permitted a range of ternary compositions wherein directional solidification produces a regular eutectic microstructure. The system was also of interest because of its potential use for high temperature turbine blade applications (Ref. 2). The particular composition chosen contained 6.2 w/o Al, 31.5 w/o Mo, balance Ni. The microstructure of the directionally solidified ingots was that of substantially square molybdenum rods surrounded by gamma prime (Ni_3Al), which in turn was separated by a small amount of gamma (nickel-aluminum solid solution) containing fine precipitated gamma prime.

Experiments were performed to examine the role of thermal gradient magnitude at the solidifying interface on the geometrical regularity of the microstructure. The difficulty in measuring the thermal gradient in the melt using superheat temperatures as high as 1800°C resulted in the examination of three liquid temperature gradient ranges (i.e. 50 to 80°C/cm, 250°C/cm, and 370°C/cm). Examination of the microstructure from directionally solidified ingots using transverse sections indicated that the highest gradient runs produced microstructure of no greater regularity than runs made at lower gradients. It was concluded that for the Ni-Al-Mo monovariant eutectic system, the magnitude of the thermal gradient at the solidifying interface is not a controlling parameter with respect to

the geometrical regularity of the microstructure, provided it is sufficient to satisfy the gradient/rate (G/R) ratio needed to produce a planar interface growth. Cusps in nominally planar liquid-solid interface adjacent to grain boundaries did not appear to cause the molybdenum rods to bend; rather, the result was to broaden rods adjacent to the grain boundary into blades along preferred interfacial crystallographic planes, which were co-parallel to the rods.

Experiments carried out previously under this contract using the $Al-CuAl_2$ eutectic, the $Al-Al_3Ni$ eutectic, and the Pb-Sn eutectic indicated that close control over the processing variables of solidification rate, thermal gradient, interface curvature, and perturbations in these parameters during the process was required in order to obtain eutectic microstructure of great regularity. In those studies, solidification rate and interface curvature were determined to be important, but not controlling, with regard to microstructural regularity.

Since the work with the Ni-Al-Mo system reported herein implies that the magnitude of the thermal gradient at the interface is also not a controlling parameter, the conclusion must be drawn that fluctuations in thermal conditions at the solidifying interface are controlling, if in fact geometrical perfection may be improved over that which has been obtained. Malmajac (Ref. 3) supports the present conclusion in that results are reported of highly regular microstructure obtained in the Cu-Al system using a solidification apparatus specifically designed to suppress thermal fluctuations which perturb the solidifying solid-liquid interface.

INTRODUCTION

NASA programs (Ref. 4) have been directed toward exploring the nearly weightless environment provided by orbiting spacecraft to conduct experiments which will lead to manufacturing products in space for use on earth.

It has been recognized that unidirectionally solidified eutectic compositions constitute a class of materials with a high degree of thermal stability which should be examined in a study of the effects of zero-gravity on solidification. Only a few of the many eutectic compositions which have been identified and subjected to unidirectional solidification have found potential uses. It is possible that with improved control over the regularity of the phase distribution in a eutectic, additional uses would be found. The role which zero-gravity processing could play in obtaining improved microstructures is presently in question as directional solidification facilities adequate to assess the effect of gravity on eutectic microstructure are not presently available.

In order to define the conditions for a zero-gravity solidification experiment which will provide insight into the role of gravity in affecting the regularity of the eutectic microstructure, an investigation of earth based (one-g) processing of unidirectionally solidified eutectics has been undertaken. The initial study evaluated various aspects of eutectic solidification resulting in a narrowing of the investigation to a study of the processing parameters which control the perfection of the eutectic microstructure. Binary eutectics in the systems Cu-Al, Ni-Al, and Pb-Sn were used as model materials which could be processed at temperatures below 1000°C. The results of the investigations made using these materials has been previously reported (Ref. 1).

It was concluded from these studies that the degree to which geometrical perfection of the microstructure in an eutectic could be produced depended on control over the growth rate, thermal gradient, and solid-liquid interface curvature. The characteristic spacing of microstructural elements is proportional to the square root of the growth rate; changes in thermal gradient produce changes in heat flow and thus in position of isotherms, one of which is the solid-liquid interface. A curved interface requires microstructural elements to be generated or terminated, leading to faults. A solidification interface propagating through an ingot subject to fluctuating control parameters is much more likely to have faults than one which proceeds under constant conditions. It was further concluded that fluctuations in these processing variables should be low compared to the growth rate. It was recommended that a study of the effects of thermal gradient be undertaken on a material of current interest for high temperature applications.

The additional study reported herein was undertaken using the ternary system Ni-Al-Mo using the composition, by weight, 31.5% Mo, 6.2% Al, balance Ni. This system was chosen because of its potential use as a high temperature turbine blade material (Ref. 2) and examined for influence of the thermal gradient in the liquid during directional solidification on the regularity of the microstructure.

Microstructural regularity was assessed using computer programs previously developed under this contract which produce nearest neighbor distributions in distances and angles. A high degree of regularity results in distinct peaks in these distributions; the less regular the structure, the more uniform the distribution of neighbor distances and angles.

EXPERIMENTAL PROCEDURES

Master melts of the Ni-6.2 w/o Al-31.5 w/o Mo alloy were made in new recrystallized alumina crucibles in a Heraeus vacuum induction melting furnace powered by a 30 kW motor generator and pumped by a 25.4 cm (10 in.) vacuum system. The system was exhausted to approximately 10^{-5} torr (0.001 N/m^2) and then backfilled with high purity argon to provide a dynamic 200 liters/hr inert cover at atmospheric pressure. Power to the furnace coil was slowly increased until melting of the nickel and molybdenum charge material was achieved. Aluminum was then added separately to the melt. Subsequently, the melt was held at a constant temperature of 1400°C for a 15-20 min homogenization period prior to pouring into copper chill molds.

Each resulting cast alloy bar was directionally solidified vertically within a nominally 0.95 cm (3/8 in.) diameter 99.7% recrystallized alumina cylindrical closed one-end tube whose wall thickness was approximately 0.2 cm. Vertical controlled solidification was accomplished in a high gradient apparatus. In this technique a known mass of alloy, typically 200 grams, contained in a one end closed cylindrical alumina crucible was positioned within the induction coil and graphite susceptor, water spray ring, and constant water level tank. With the water spray inpinging on the crucible and the water tank full, melting was accomplished by inductively coupling to the stationary graphite sleeve which provided radiant heating to the crucible. Power requirements were established by monitoring the temperature at the top of the melt with an optical pyrometer while simultaneously measuring the temperature within the melt using a tungsten-5% rhenium/tungsten-26% rhenium thermocouple. Successful measurements of gradients of 375°C/cm and 80°C/cm at the solid-liquid interface were made. These data are shown in Fig. 1. Thermal gradients were estimated for other runs from the monitored melt temperature. Controlled freezing commenced by the withdrawal of the Al_2O_3 crucible through the water spray ring at 1.5 cm/hr (4.17μm/sec). Excluding small end-affected regions, the rate of freezing in this setup has been found to be equal to the velocity of crucible withdrawal.

Differential thermal analysis of this alloy composition showed the solidus temperature to be 1309°C and the liquidus temperature to be 1310°C.

Assessment of the microstructural regularity was accomplished by establishing the coordinates of the centers of the molybdenum rods with respect to an arbitrary origin using an automatic coordinate generating table. The (x,y) values for each point were directly stored in memory in the Research Center's PDP-6 computer. A program developed previously under this contract (Ref. 2) generated a histogram from the data which represented the distribution of neighbor distances up to 12.5 microns (50 millimeters at 4000 magnification)

using photographs of transverse sections. This corresponds to evaluation of distances to first, second, and third nearest neighbors in an hexagonal array. The program also computes the angles of the six nearest coordinates to a given origin and compiles these into a histogram. A regular array will generate distinct peaks in these histograms representing the average distances to first, second, and third nearest neighbors, and the average number of first nearest neighbors found in 5° angular increments relative to the origin point. A uniformly (random) distribution of rods generates distributions in neighbor distances and angles which have no peaks.

Seeding experiments were undertaken to obtain directionally solidified ingots having one or at most a few grains. The first method used consisted of directionally solidifying an ingot, centerlessly grinding it to reduce the diameter so that remelting would not break the crucible, placing it tail first (tail = final solidification end of ingot) in a new crucible, and performing a second directional solidification. The second method made use of dip coated Al_2O_3-SiO_2 shells produced by PWA's Experimental Foundry. The base coat slurry was rich in alumina which is compatible with the Ni-Al-Mo system. The tapered shell crucibles were approximately 1.3 cm inside diameter by 15 cm long with the closed end tapered to a seed rod holder 0.015 cm diameter by 2 cm long. Seed rods were prepared for these crucibles by choosing large grains from directionally solidified ingots 2 cm long and using EDM techniques to obtain a single grain section. Three such seed rods were prepared. Directional solidification experiments were performed using three tapered crucibles in the same high-gradient apparatus used for the runs in the recrystallized high purity alumina crucibles.

RESULTS AND DISCUSSION

The directional solidification of the Ni-6.2Al-31.5Mo alloy produced ingots with square molybdenum rods spaced 2-4 μm between centers on the average in a gamma/gamma prime matrix. The crystallographic orientations of the interpenetrating molybdenum bcc and gamma/gamma prime fcc lattices are described in Ref. 2 as rotated about a mutual [100] growth direction by 45 degrees. Further, the gamma and gamma prime lattices were semi-coherent and the {110} planes of molybdenum and the {100} cube planes of gamma prime were found to form the square interfaces of the rods.

In the course of this investigation, 17 ingots were directionally solidified. Four of these were used for seeding experiments; the remaining 13 runs are listed in Table I with estimated thermal gradients.

Each ingot was transversely sectioned and metallographically polished in at least two regions, generally 2 to 5 cm apart, starting at 3 to 5 cm from the head end (initial freezing end), for examination of the microstructure. Among the ingots the gradient varied by a factor of 4 (80°C/cm to 375°C/cm); the microstructure of the very low gradient runs tended to cellularity but did not appear significantly different at the higher gradients except for the lack of cellularity. The phenomenon of banding was observed to occur in many of the ingots; while this did not seem to significantly affect the appearance of the microstructure, it indicated that thermal fluctuations must be occurring during the solidification.

Typical grain structure in transverse sections of the ingots is shown in Figs. 2 and 3. These ingots were processed in thermal gradients of 80°C/cm (A76-225), 50°C/cm (A76-398), 370°C/cm (A76-308), and 325°C/cm (A76-402). Ingots A76-398 and -402 were melt-back experiments; the low gradient ingot A76-398 appears to have far fewer grains initially than the higher gradient A76-402 at 3 cm from the head end, but become comparable at the 5 cm section. It is not clear why the melt-back ingot processed at low gradient should have fewer grains initially than the high gradient ingot, but in any case, neither melt-back experiment yielded an ingot which was single grained or had only two or three grains.

Typical microstructure observed in transverse sections in the ingots produced are shown in Figs. 4 and 5. Figure 4 compares A76-225 and the repeat, melt-back run A76-398. These runs were low gradient, of the order 50 to 80°C per cm. A large portion of the rods are broadened into blades in a pattern suggestive of an approach to a cellular structure. Figure 5 compares A76-308

and its repeat, melt-back run A76-402. The gradients at which these ingots were processed was between 350 and 370°C per cm. The melt-back ingots appear to have slightly better microstructure than the single run ingots they repeat. The microstructure shows no signs of cellularity, and the regularity of arrangement of the molybdenum rods, while acceptable, is no better than that shown in Fig. 6 for A76-131, processed at an estimated 250°C/cm thermal gradient. Thus, increasing the gradient from 250 to 375°C/cm does not appear to result in improved microstructural regularity.

A small section of a large grain of ingot A76-131 was electropolished and replicated. An electron microscope picture of the replication at a magnification of 8700 is shown in Fig. 7. In such a small section, the regularity of the structure appears excellent, but in larger area units becomes more randomized, especially in angle distribution of nearest neighbors. Selected small area sections such as Fig. 6 show much greater regularity than the average over large areas. Figure 8 shows the distribution in nearest neighbor distances obtained from a selected small area of ingot A76-131 (G_L = 250°C/cm), the same ingot which yielded the relatively regular rod distribution of Fig. 6. The distribution for the selected small area of A76-225 (G_L = 80°C/cm) is shown in Fig. 9 for the first nearest neighbors. The distribution from A76-131 (Fig. 8) has a sharp peak giving the average distance between rod centers as 2.4 micrometers. The distribution from the selected area of A76-225 (Fig. 9) gives peaks indicating a first nearest neighbor average distance of 3.25 micrometers. Thus, selecting small areas of transverse sections provides a means of analyzing local regularity, but this is not characteristic of the section as a whole. Figures 10 and 11 present nearest neighbor spatial and angular distributions which are typical of large areas in transverse section. These lack peaks which can be related to geometrical regularity, thus indicating that on a large scale, the structure would be described as random with regard to the probability of finding rods at a given distance apart above a minimum, or at a specific angle relative to a fixed axis.

Another seeding experiment was undertaken using molds made by dipping wax preforms into ceramic slurry and then into coarser grit. The shells are dewaxed and heated to yield molds with acceptable strength for handling and subsequent use. These molds were made to accept a 0.15 cm by 2 cm long seed rod in the head end. This portion of the mold then tapers at about 45° to a 1.3 cm diameter by 15 cm long ingot section. Seed crystals were cut from large grains in directionally solidified Ni-6.2Al-31.5Mo ingots by EDM techniques. These were inserted in the seed-holder portion of the foundry molds, and cast material placed on top of them. The molds were positioned in the D.S. apparatus so that part of the seed portion was impinged by water from the spray ring. Three attempts were made to produce directionally solidified ingots; the first

run did not permit melt back into the seed rod, so that the seed was not effective. The second and third runs broke out of the crucibles. Of the latter two, the second run broke out part way through the run, but the third run broke out on start-up, so that no material was saved. Figure 12 shows the microstructure obtained in the first run where the seeding was not effective. This ingot was processed at a low gradient; its microstructure is similar to that of ingots solidified in the recrystallized aluminum oxide crucibles at high gradient which yielded good microstructure. Figure 13 shows the dendritic structure obtained in ingot A76-506 wherein the seed was effective, but the run was not completed. A macro view of this ingot did not exhibit any structure which could be described as a grain boundary, as illustrated in Fig. 14.

CONCLUSIONS

Optical evaluation of transverse sections as well as computer analysis of nearest neighbor distance and angular position distributions permitted the following statements with regard to the effect of thermal gradient on producing regular microstructure:

1. Low gradient (G_L = 50 to 80°C/cm) processing produced some ingots with areas of regular microstructure but generally many areas around grain and subboundaries had rods broadened into blades, indicative of an approach to cellular growth.

2. High gradient (G_L = 250 to 375°C/cm) processing produced ingots with areas of regular microstructure, with no indication of cellular growth.

3. As much variation was observed in the regularity of rod structure among ingots processed at high gradients as was observed among ingots processed at low gradients.

It was concluded that increasing the magnitude of the thermal gradient above the level required to assure plane front growth does not ipso facto improve the geometrical regularity of the microstructure in the Ni-6.2Al-31.5Mo ternary studied.

Considering the processing variables of solidification rate, thermal gradient at the interface in the liquid, interface curvature, and the time stability of these, this study has fixed the solidification rate (1.5 cm/hr or about 4 μm/sec), varied the thermal gradient in the liquid from 50°C/cm to 375°C/cm, and accepted the interface curvature these conditions produced. Examination was made near the axis of the ingot to ensure the least curvature. Stability of the processing variables reflects the stability of the mechanical systems used for traverse and the fluctuations which may occur in the power supplied by the 35 kW Lepel induction generators. Small power fluctuations are damped by the graphite susceptor, but the thermal mass of the system is limited so that some fluctuations will be seen by the system. Bands, which are microstructural irregularities caused by the system response to some thermal fluctuation, occurred in the ingots processed. Fluctuations in the steady state process of solidification do occur occasionally. Subtler compositional fluctuations than those which cause bands observable by light microscopy may be postulated. Impurities may be responsible for subtle surface energy changes especially near high angle grain boundaries but their quantification was difficult if not impossible to measure (Ref. 5).

With regard to the effects of eliminating thermal fluctuations at the solidifying interface, the work reported by Malmajac (Ref. 3) implied that elimination of thermal fluctuations resulted in a large decrease in fault density in certain eutectic systems. A solidification furnace was described which consists of three zones with a sophisticated temperature control system which permitted solidification of the copper-aluminum eutectic in a vertical traverse system resulting in regions up to 20 mm long (in longitudinal section) with perfect, fault-free microstructure. Convection was minimized, if not eliminated, by solidifying ingots vertically from the bottom up, as done by Malmajac and in the work reported herein as well.

The reduction in thermal fluctuations attained by Malmajac was seen as the causative factor in the reduction of faults he obtained in the Cu-Al eutectic. It is suggested that stability of the thermal conditions providing the gradient in the liquid was more important than the magnitude of the gradient in terms of producing highly regular microstructure.

RECOMMENDATIONS FOR SPACE PROCESSING OF EUTECTICS

The work carried out under this contract as a whole suggests that in planning eutectic solidification studies in a low gravity environment, a high degree of stability must be maintained over the thermal conditions at the solidifying interface. Actual magnitudes of solidification rate and thermal gradient may be any combination which satisfies plane-front growth conditions as established by ground-based tests.

Development of directionally solidified materials in thin film form may provide interesting structures. Experiments (Ref. 1) showed that lead-tin eutectic films of the order of 20 micrometers thick solidified with lamellae which spanned the thickness of the film without faults.

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2. F. D. Lemkey, Final Report, Contract N62269-75-C-0129, Naval Air Development Center, December 1975.

3. Y. Malmajac, "Controlled Growth of Al-Al_2Cu Eutectic Crystals and Improvement of Their Morphological Stability", Proceedings of In Situ Composites Conference (CISC II), Bolton Landing, New York, Sept. 1975, edited by M. Jackson, J. Walter, R. Hertzberg, and F. Lemkey, Xerox Individual Publishing Co., Lexington, MA (1976).

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5. F. D. Lemkey, Final Report, Contract N62269-76-C-0107, December 1976.

Table I

Run # A76-	Rate cm/hr	Gradient °C/cm	Max Temp °C (by Optical Pyrometer)	Remarks
102	3	250(est)	1690	Near cellular
131	1.5	250(est)	1650	Good
225	1.5	80(meas)	1400	Near cellular
285	1.5		control: 1650	Resistance furnace
291	1.5	375(est)	1850	Power failure @ 6 cm
308	1.5	370(meas)	1850	Power failure @ 6 cm
398	1.5	50(est)	1330	Melt-back; repeat 225
402	1.5	325(est)	1770	Melt-back; repeat 308
434	1.5	350(est)	1800	Melt-back; repeat 308
437	1.5	350(est)	1790	Melt-back; repeat 308
502	1.5	50(est)	1340	*Seeded; foundry crucible
506	1.5			*Seeded; foundry crucible
514	1.5			*Seeded; foundry crucible

*melt broke out of crucible before traverse

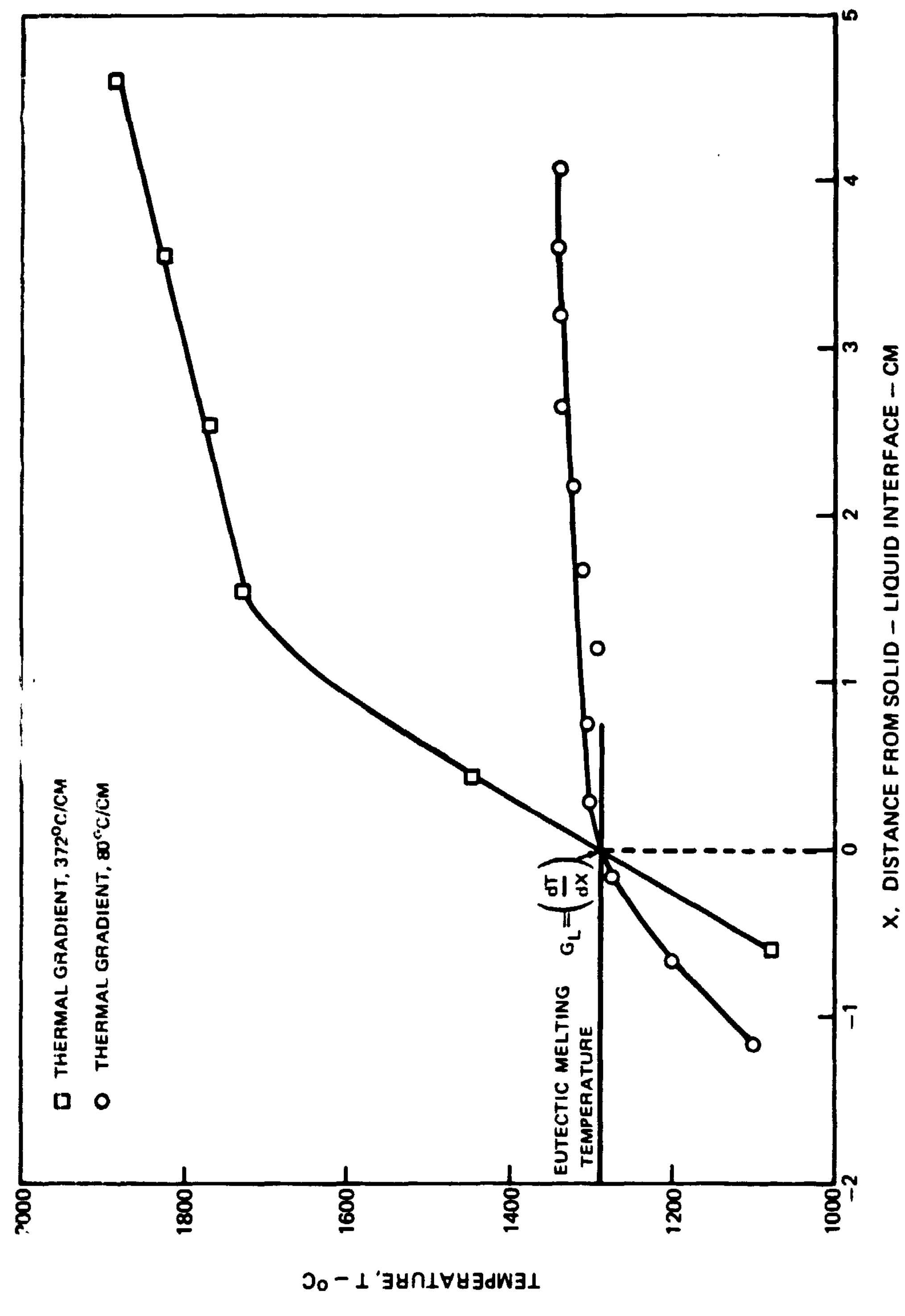
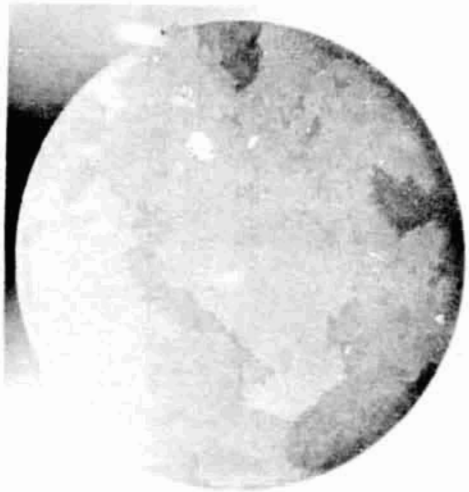
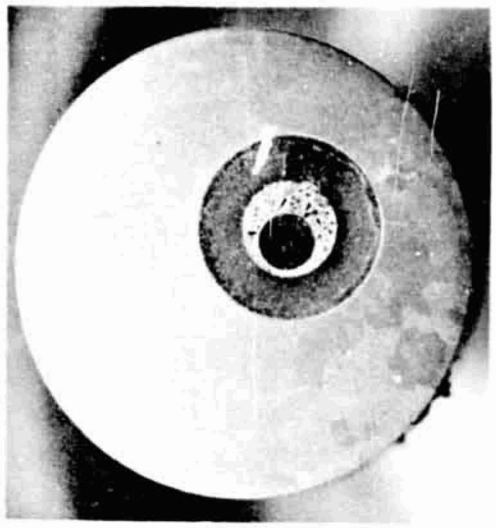


FIG. 1 Thermal Profile Within Ni–6.2 Al–31.5 Mo Eutectic During Directional Solidification at 1.5 cm/hr



6.4x

A76–225 5 CM FROM HEAD

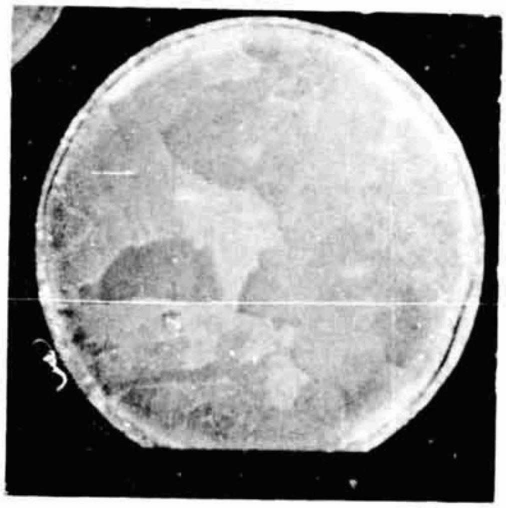


6.4x

A76–225 10 CM FROM HEAD

$G_L = 80^{o}$ C/CM

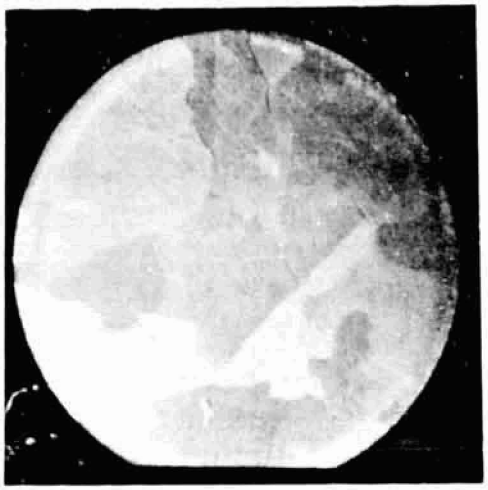
A76–398 3 CM FROM HEAD 6.4x



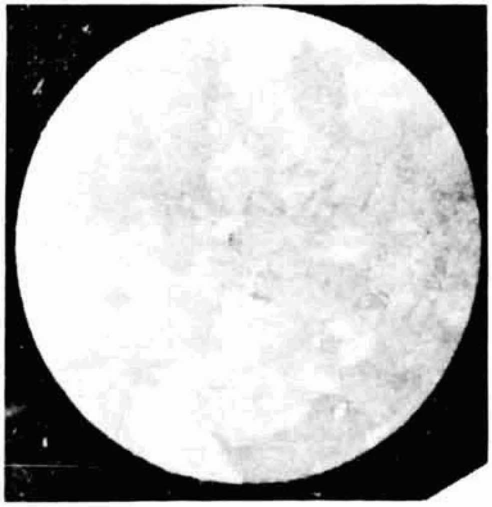
A76–398 5 CM FROM HEAD 6.4x

$G_L = 50^{o}$ C/CM

Fig. 2 Grain Structure of Ni – 6.2 Al – 31.5 Mo

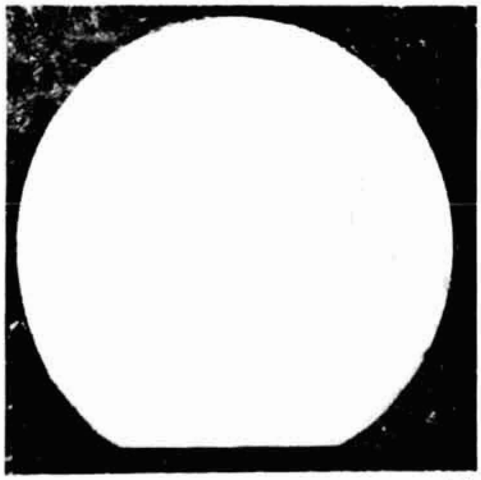


A76–308 3 CM FROM HEAD 6.4x

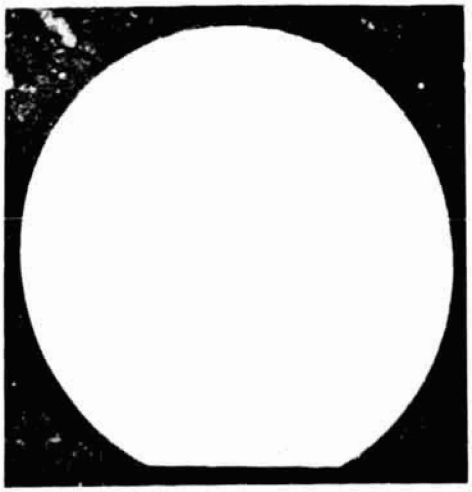


A76–308 8 CM FROM HEAD 6.4x

$G_L = 370^o$ C/cm



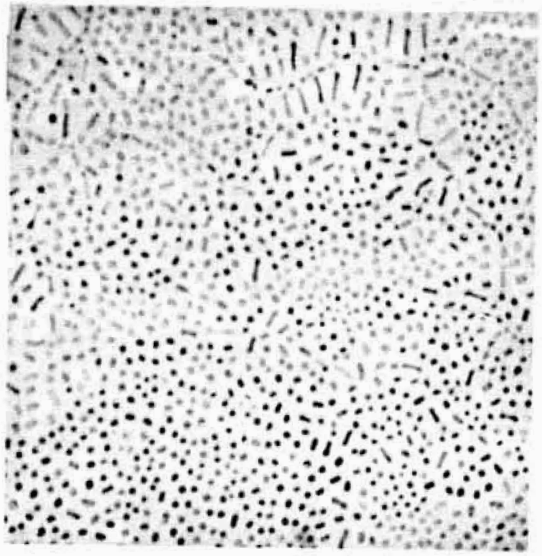
A76–402 3 CM FROM HEAD 6.4x



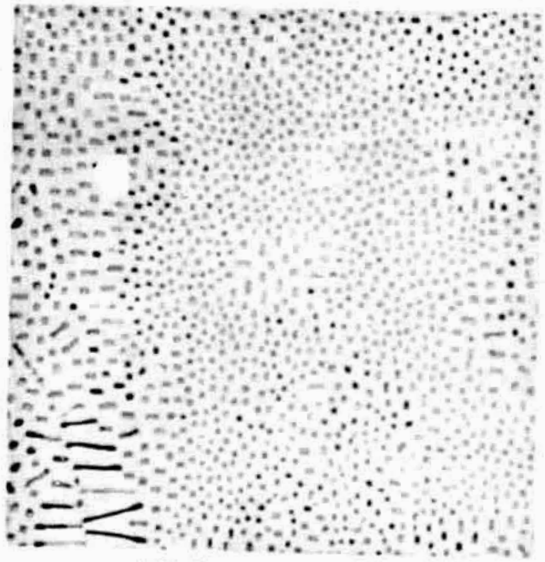
A76–402 5 CM FROM HEAD 6.4x

$G_L = 325^o$ C/cm

Fig. 3 Grain Structure of Ni – 6.2 Al – 31. Mo Ingots



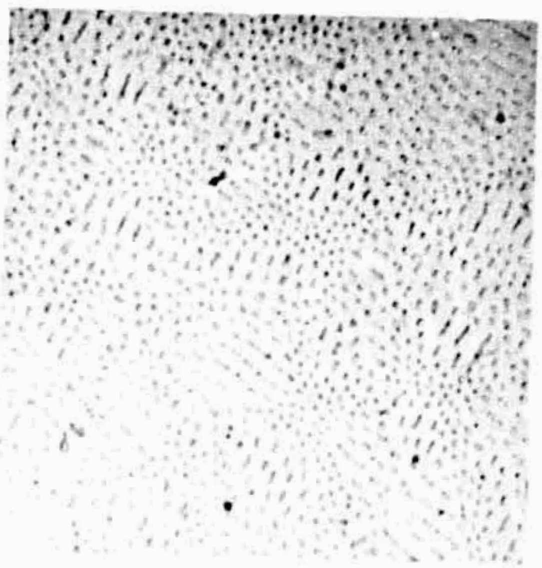
A76–225 5 CM FROM HEAD 10μ



A76–225 10 CM FROM HEAD 10μ

G_L = 80° C/CM

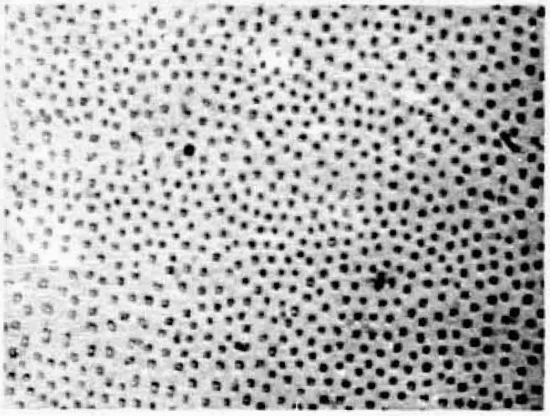
A76–398 AT 3 CM FROM HEAD 10μ



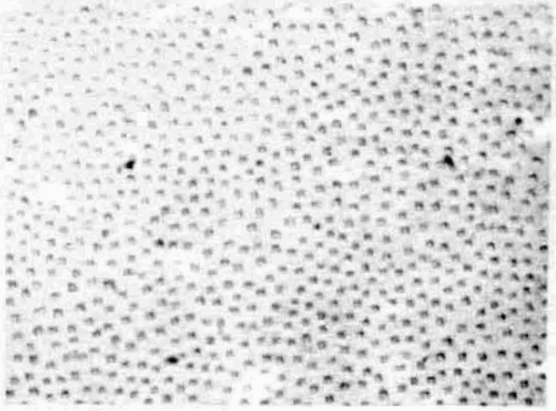
A76–398 5 CM FROM HEAD 10μ

G_L = 50° C/cm

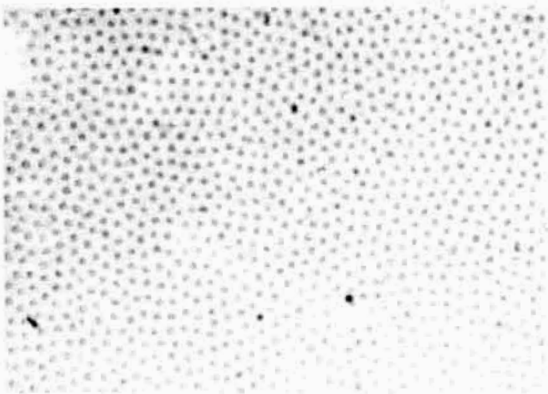
Fig. 4 Microstructure of Ni – 6.2 Al – 31.5 Mo Ingots



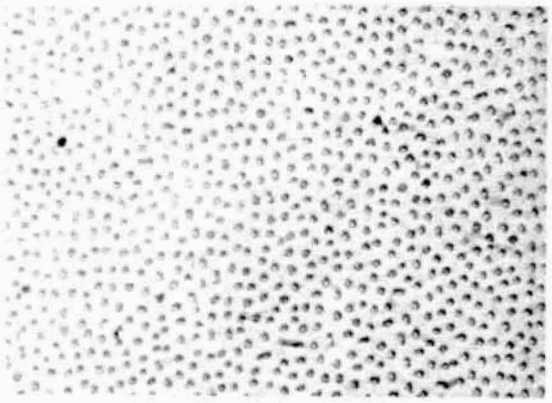
A76–308 5 CM FROM HEAD 10μ



A76–308 8 CM FROM HEAD 10μ



A76–402 3 CM FROM HEAD 10μ



A76–402 5 CM FROM HEAD 10μ

GL 325°C/cm CM

Fig. 5 Microstructure of Ni–6.2 Al–31.5 Mo Ingots

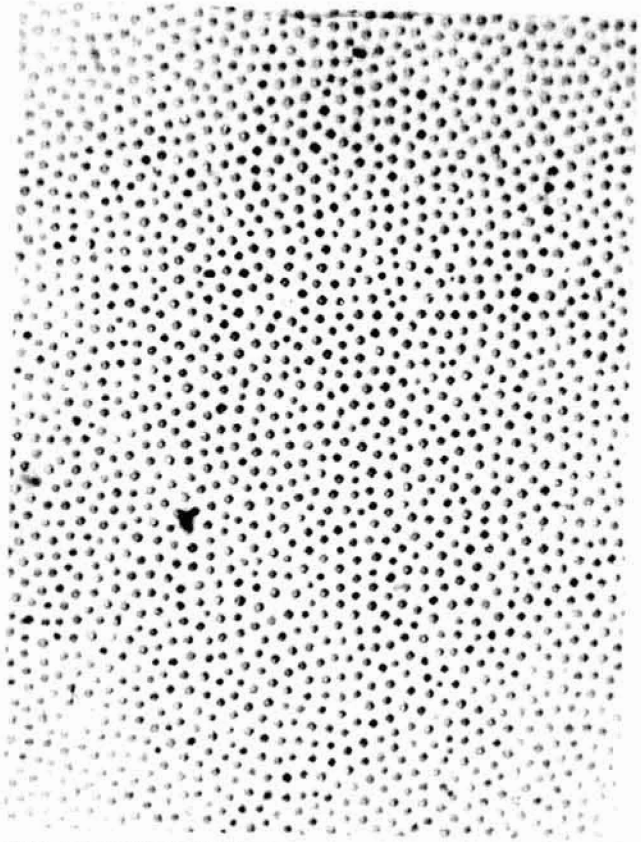


Fig. 6 Microstructure of Ni–6.2Al–31.5 Mo Ingot Solidified with a Gradient of 250°C/cm

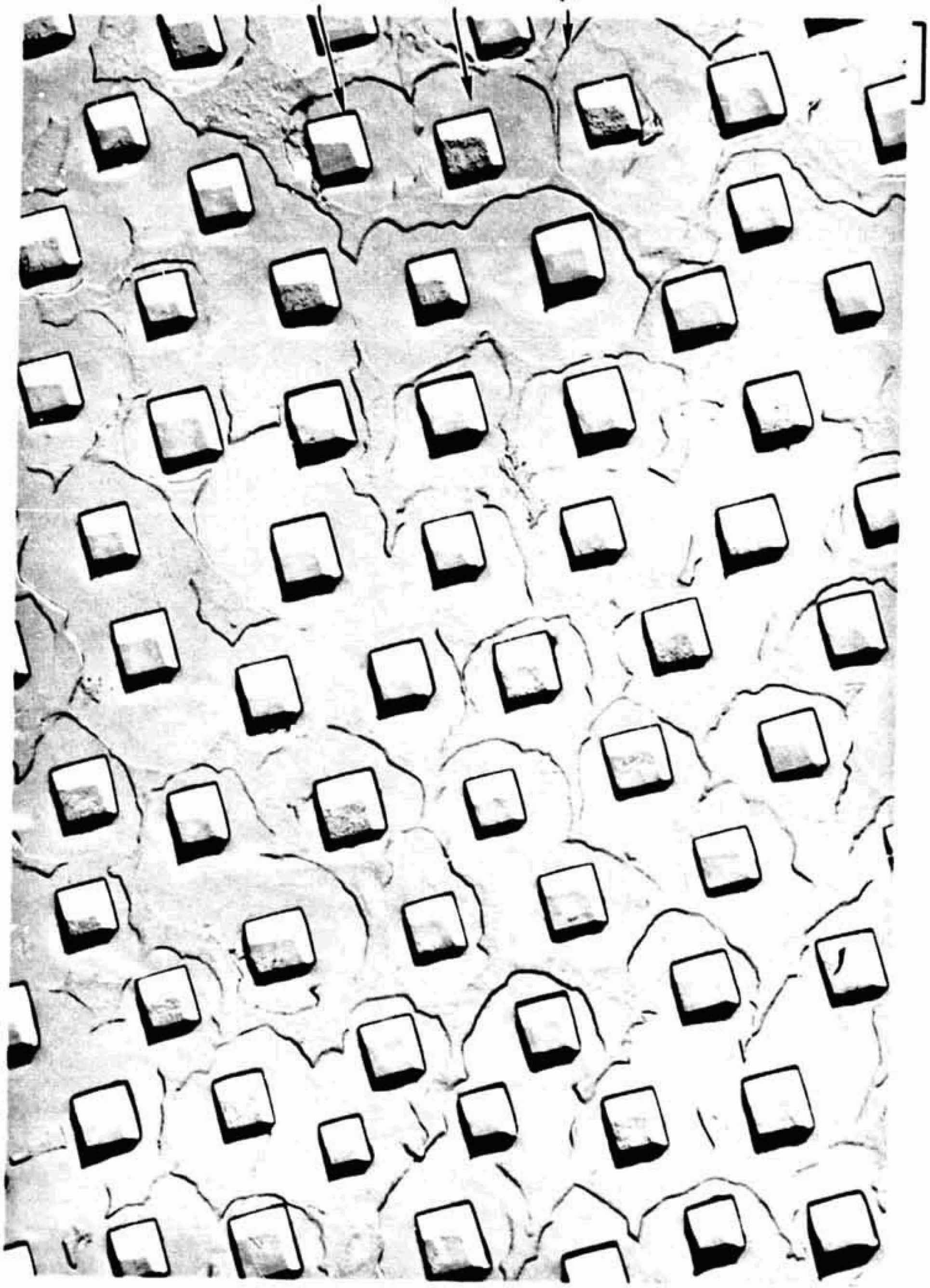


Fig. 7 Typical Structure in Small Area of N_i – 6.2 Al – 31.5 Mo Ingot

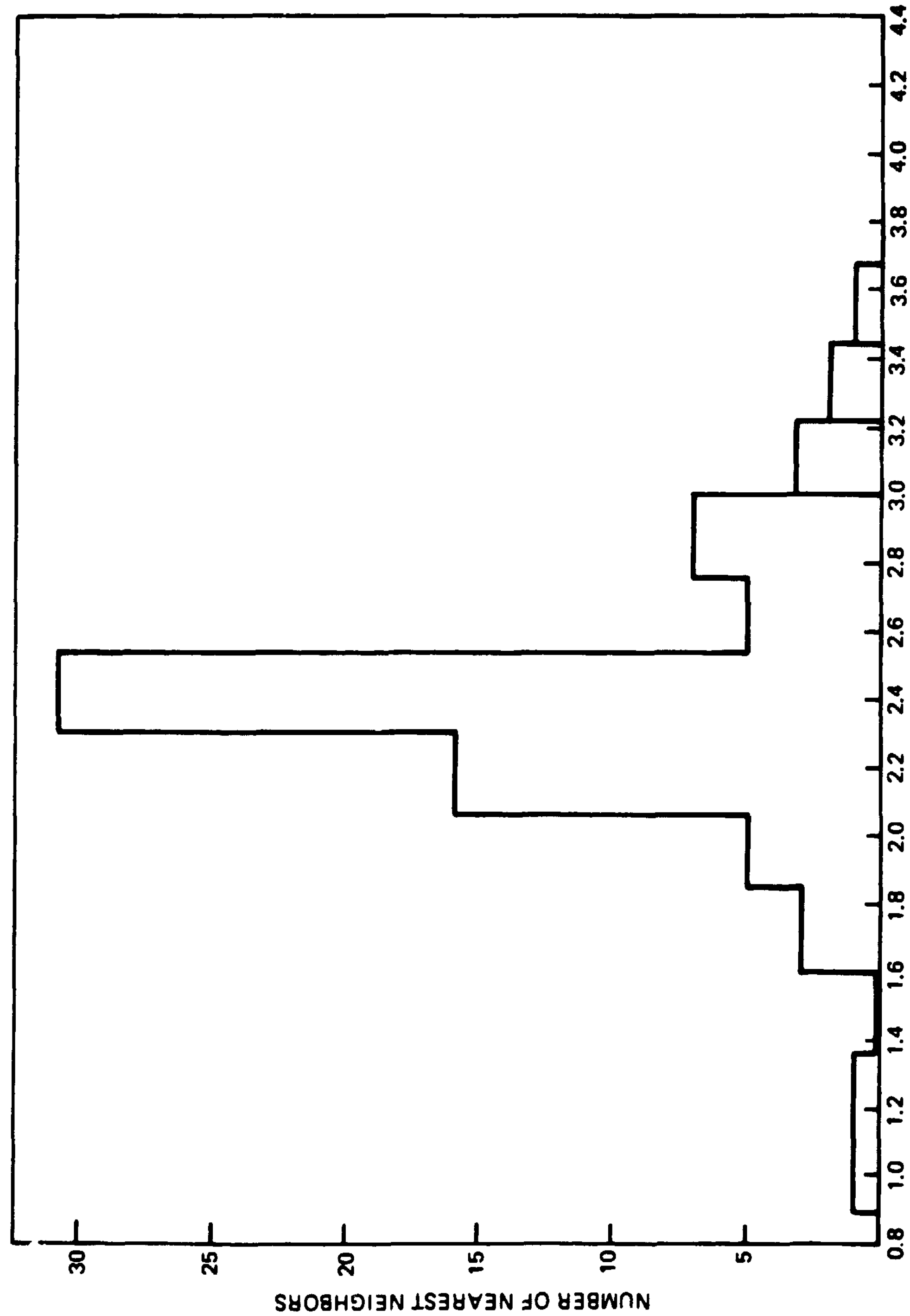


Fig.8 Distribution of First Nearest Neighbors for Small Area Specimen A76–131,Ni–6.2Al–31.5Mo

R77-911721-11

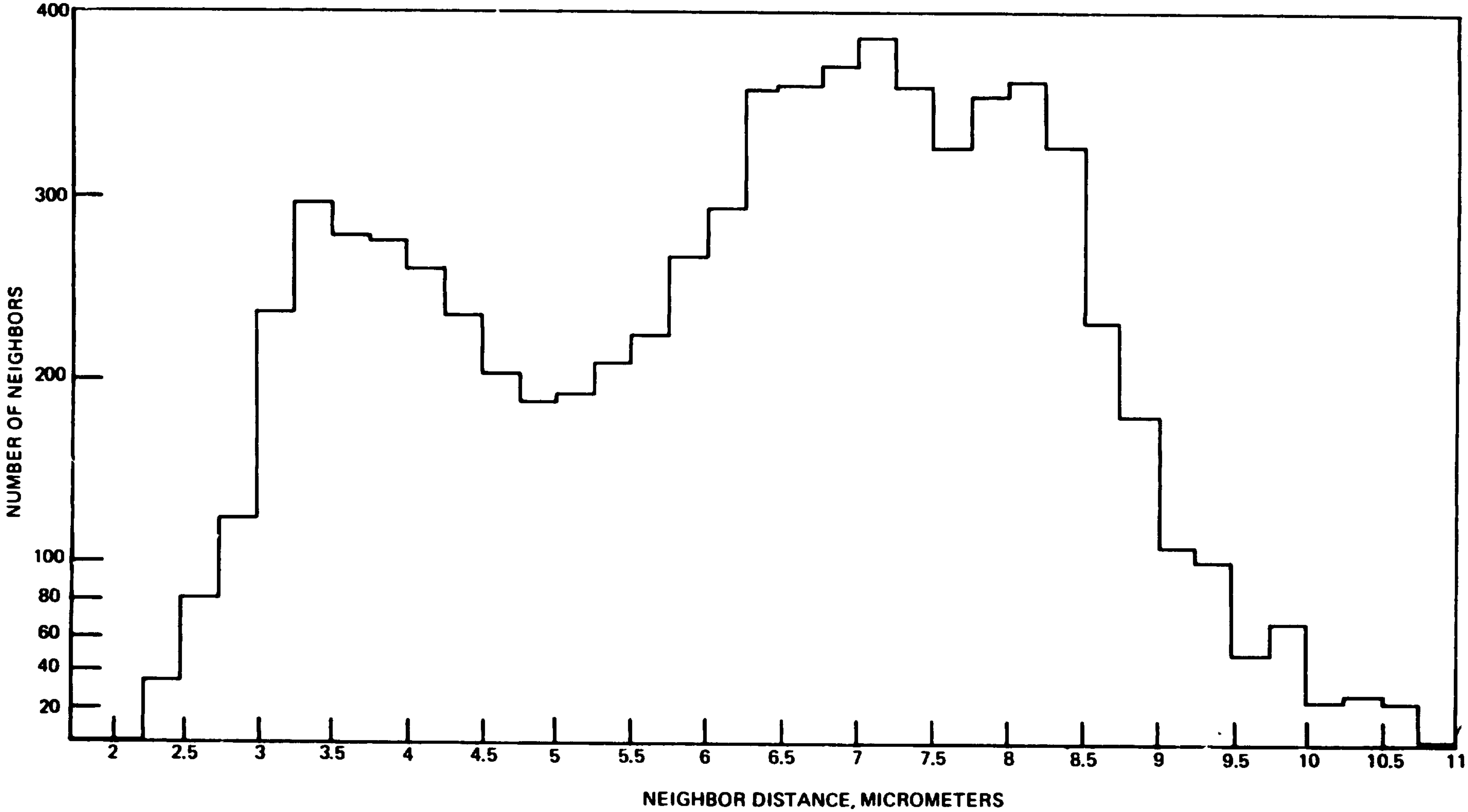


Fig. 9 Nearest Neighbor Distribution for Ni – 6.2 Al – 31.5 Mo D. ... en (Small Area)

77-01-50-3

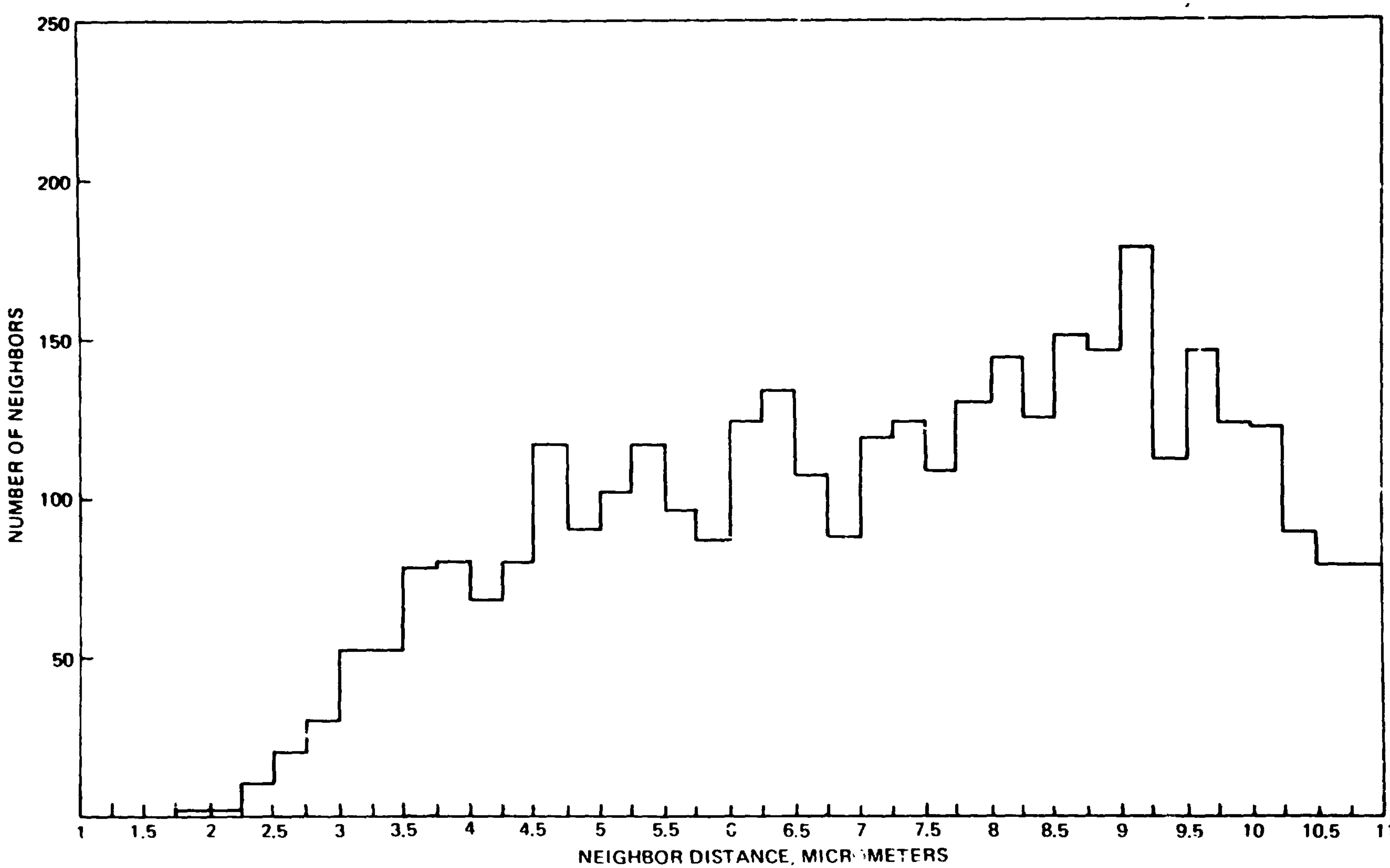


Fig. 10 Nearest Neighbor Distribution for Ni – 6.2 Al – 31.5 Mo D.S. Specimen A76–225–5

77-01-50-2

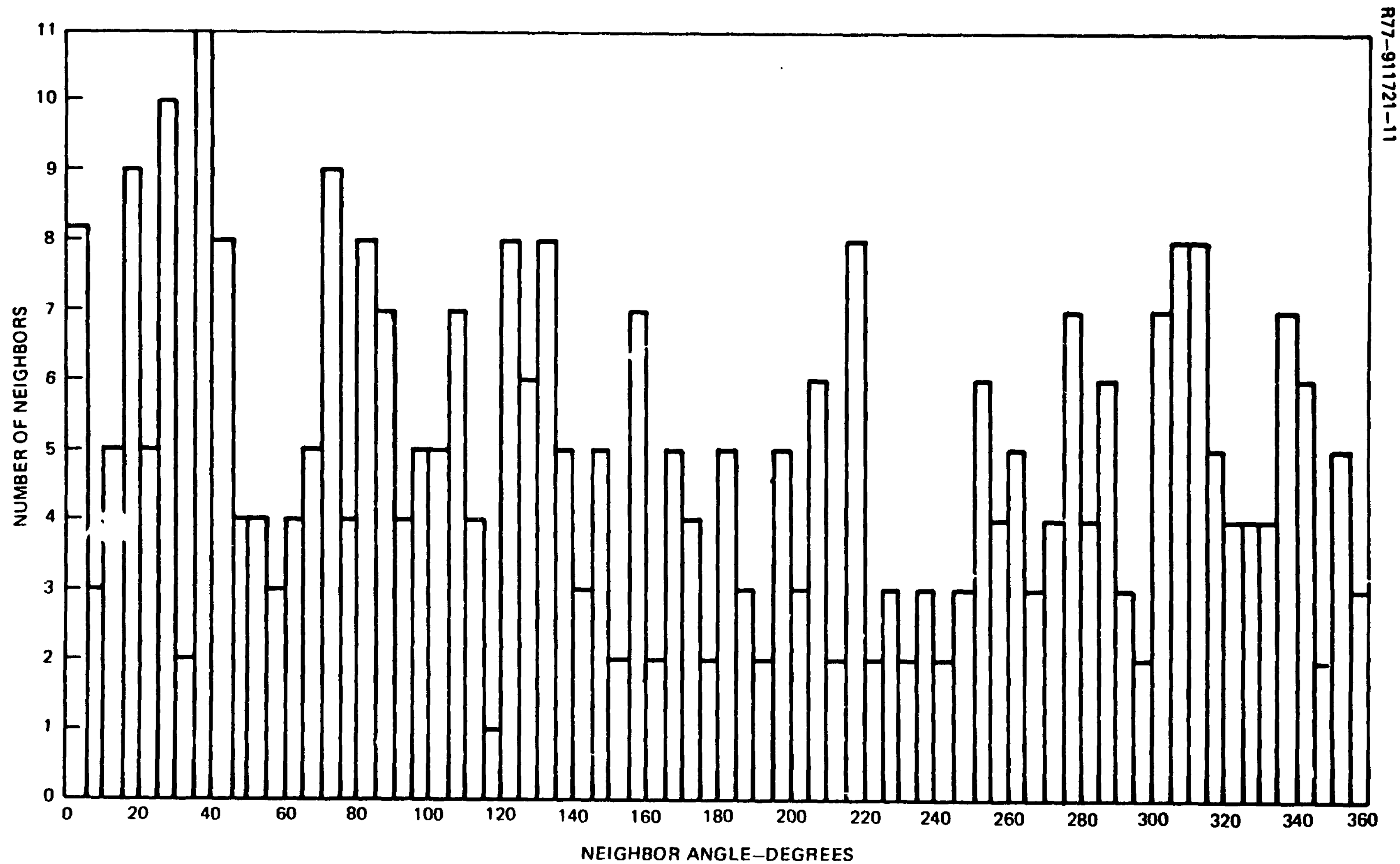


Fig. 11 Nearest Neighbor Angle Distribution for Ni–6.2Al–31.5Mo Specimen

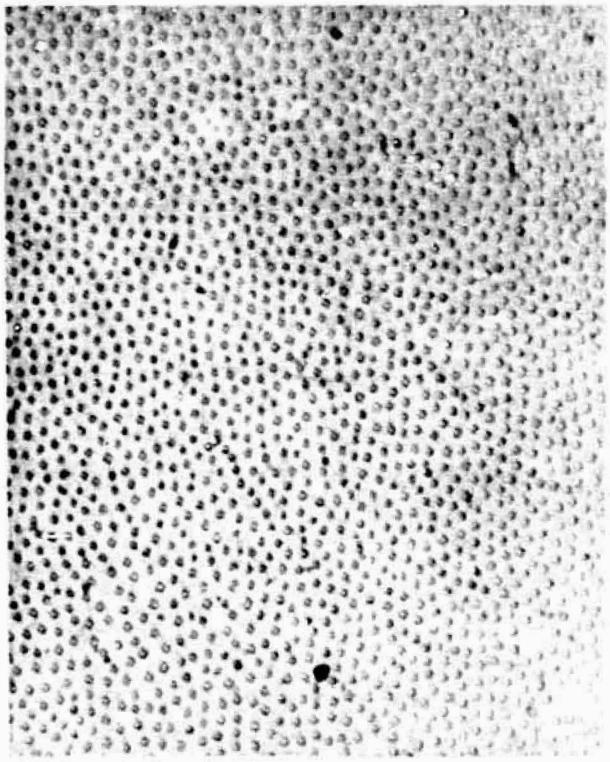


Fig. 12 Typical Microstructure of Ingot A76–502 (Seeding Not Effective)

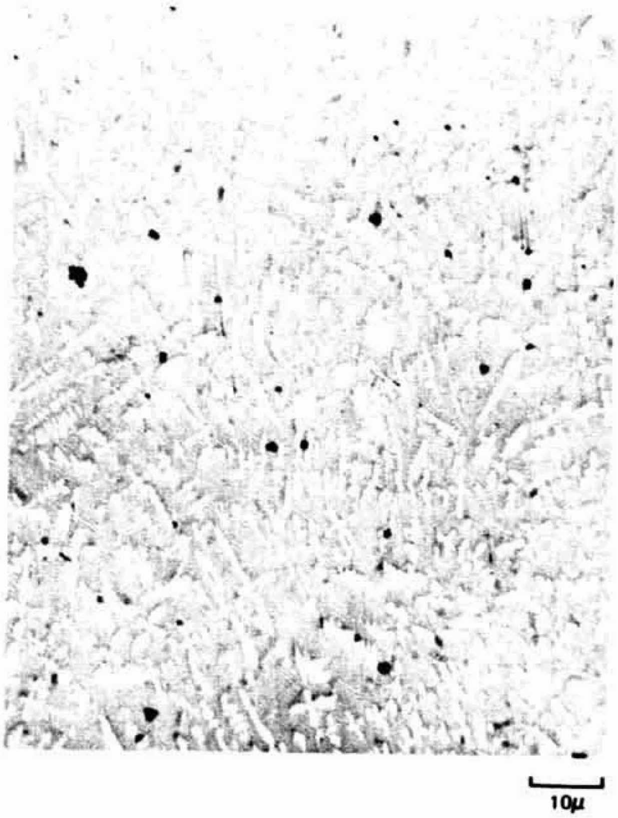


Fig. 13 Dendritic Microstructure Developed in Head End of Seeded Ingot A76–506
(Run not completed)

4.25X

Fig. 14 Composite Crucible Seeded Ingot A76-506–Macroview